Application Note 1105

Determination of Anions and Cations in Produced Water from Hydraulic Fracturing

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Key Words
Fracking, Capillary IC, Dionex ICS-5000+, Dionex ICS-2100, Dionex IonPac CS16, Dionex IonPac AS18

Goal
Quantify anions and cations in produced water from hydraulic fracturing using ion chromatography.

Introduction
Hydraulic fracturing (also known as "fracking") is being used extensively in the U.S. and its potential for hydrocarbon recovery is beginning to be explored around the world in places such as the UK and China.[1,2] Fracking consists of drilling a well vertically down several thousand feet (thousands of meters) to a layer of hydrocarbon-rich shale and then horizontally for a mile (1.6 km) or more. Fracturing fluid is then injected under high pressure through perforations in the horizontal well casing to fracture the adjacent shale, releasing the natural gas and oil trapped there. The liquid portion of hydraulic fracturing fluid is composed of approximately 99% water with the remainder consisting of chemical additives. Sand is added to this fluid as a proppant to keep open the cracks that are formed, thereby facilitating oil and gas recovery. To optimize recovery, additives are tailored to the site's geology and the chemical characteristics of the water used.[3] Additives include friction reducers, scale inhibitors, anti-bacterial agents, and corrosion inhibitors.[4] Following the release of pressure, the fluid that returns to the surface is referred to as flowback water, which is then pumped into lined storage ponds or tanks prior to recycling or disposal. Once gas or oil appears, which marks the start of production, the fluid recovered is called produced water. This water will contain some residual fracking fluid, but will consist primarily of water that was present within the shale layer prior to fracturing (formation water). While fracking wastewater (flowback and produced) can be disposed of by injection into disposal wells, these waters are increasingly being reused to reduce the demand on local water resources and to decrease wastewater management costs.[3]

Flowback wastewater can contain high concentrations of anions such as ~90,000 mg/L chloride, ~900 mg/L bromide, and ~2 mg/L organic acids, all of which can impact treatment or reuse.[5] If treated wastewater is used for additional fracking, the effectiveness of additives can be diminished in the presence of high salt concentrations and any metals that remain. Cations such as sodium, calcium, and strontium can also be present at high mg/L to g/L concentrations, which promotes scale formation and negatively impacts hydrocarbon recovery.[6] Knowing the wastewater's ion content is also important for determining the treatment plan prior to discharging to surface water.

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This Application Note describes the quantification of anions and cations in produced water from hydraulic fracturing sites in California (CA), North Dakota (ND), and Texas (TX) using a Thermo Scientific™ Dionex™ ICS-5000⁺ Reagent-Free™ High-Pressure™ Ion Chromatography (HPIC™) system with a Thermo Scientific™ Dionex™ IonPac™ CS16 capillary column and Thermo Scientific™ Dionex™ ICS-2100 Integrated Reagent-Free IC (RFIC) system with a Dionex IonPac AS18 microbore column. Separations were optimized to resolve all measured ions within 35 min. Knowledge of the anion and cation composition of produced water can aid in the development of water treatment and reuse strategies.

Equipment

Microbore

- Dionex ICS-2100 Integrated RFIC System,† including:
  - Isocratic Pump
  - Vacuum Degasser
  - EG Eluent Generator
  - Column Heater
  - High-Pressure 6-Port Injector
  - Conductivity Cell and CD Conductivity Detector
- Thermo Scientific Dionex EGC III KOH Eluent Generator Cartridge (P/N 074532)
- Thermo Scientific Dionex CR-ATC Continuously Regenerated Anion Trap Column (P/N 060477)
- Injection Loop, 2.5 µL

† This application can be performed on any Dionex ICS system capable of eluent generation and microbore operation.

Capillary

- Dionex ICS-5000⁺ Reagent-Free HPIC capillary system including:
  - SP Single Pump or DP Dual Pump
  - EG Eluent Generator module
  - DC Detector/Chromatography module with Thermo Scientific™ Dionex™ IC Cube™ (Capillary) and CD Conductivity Detector
- Thermo Scientific Dionex EGC-MSA Eluent Generator Cartridge (P/N 072077)
- Thermo Scientific Dionex CR-CTC II Continuously Regenerated Cation Trap Column (P/N 072079)
- Injection Loop, 0.4 µL

Autosampler

- Thermo Scientific Dionex AS-AP Autosampler with a 250 µL Sample Syringe (P/N 074306) and 1200 µL Buffer Line Assembly (P/N 074989)
- Vial Kit, 10 mL, Polystyrene with Caps and Blue Septa (P/N 074228)

Software

- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2

Reagents and Standards

- Deionized water, Type I reagent grade, 18 M -cm resistance or better

Anions

- Thermo Scientific Dionex
  - 1000 mg/L Chloride P/N 037159
  - 1000 mg/L Fluoride P/N 037158
  - 1000 mg/L Sulfate P/N 037160
- 1000 mg/L Bromide (Ultra Scientific P/N ICC-001)
- Sodium Acetate, anhydrous, ACS Grade (Fisher Scientific P/N S210)
- Sodium Formate, ACS Grade (Fisher Scientific P/N S648)
- Sodium Nitrate, ACS Grade (Fisher Scientific P/N S343)

Cations

- Ultra Scientific
  - 1000 mg/L Lithium P/N ICC-104
  - 1000 mg/L Sodium P/N ICC-107
  - 1000 mg/L Ammonium P/N ICC-101
  - 1000 mg/L Potassium P/N ICC-106
  - 1000 mg/L Magnesium P/N ICC-105
  - 1000 mg/L Calcium P/N ICC-103
- Strontium chloride, hexahydrate, ACS Grade (Fisher Scientific P/N S541)
- Barium chloride, dihydrate, ACS Grade (Fisher Scientific P/N B34)

Samples

Produced water from Texas (Eagle-Ford Shale), California (Monterrey Shale), and North Dakota (Bakken Shale).

Conditions

Anion Method (2 mm, Microbore)	
Columns:	Dionex IonPac AG18 Guard, 2 × 50 mm (P/N 060555) Dionex IonPac AS18 Separation, 2 × 250 mm (P/N 060553)
Eluent Source:	Dionex EGC III KOH cartridge
Gradient:	15 mM KOH (0–3 min), 15–29 mM KOH (3–4 min), 29 mM KOH (4–12 min), 15 mM KOH (12–17 min)
Flow Rate:	0.25 mL/min
Column Temp.:	30 °C
Inj. Volume:	2.5 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ AERS™ 500 Anion Electrolytically Regenerated Suppressor (P/N 082541), recycle mode, 18 mA
Background Conductance:	< 1.2 µS
Noise:	< 1 nS
System Backpressure:	2200 psi

Cation Method (Capillary)	
Columns:	Dionex IonPac CG16 Guard, 0.5 × 50 mm (P/N 075402) Dionex IonPac CS16 Separation, 0.5× 250 mm (P/N 075401)
Eluent Source:	Dionex EGC-MSA cartridge
Gradient:	20–30 mM MSA (0–10 min), 30–55 mM MSA (10–18 min), 55 mM MSA (18–34 min), 20 mM MSA (34–40 min)
Flow Rate:	0.010 mL/min
Column Temp.:	40 °C
Inj. Volume:	0.4 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ CCES™ 300 Cation Capillary Electrolytic Suppressor (P/N 072053), recycle mode, 11 mA
Background Conductance:	< 0.2 µS
Noise:	< 1 nS
System Backpressure:	1300 psi

Preparation of Standards

To prepare 1000 mg/L stock solutions, accurately weigh the amounts indicated in Table 1, transfer solid to a 100 mL volumetric flask, and fill to the mark with deionized water. Mix thoroughly. Concentrated standards should be stable for at least one month when stored at 4 °C.

Table 1. Mass of reagent to prepare a 100 mL stock standard.

Analyte	Chemical	Mass (g)
Acetate	Sodium acetate	0.1389
Barium	Barium chloride	0.1779
Formate	Sodium formate	0.1511
Nitrate	Sodium nitrate	0.1152
Strontium	Strontium chloride	0.3043

Working Standard Solutions

Prepare the highest concentration working standard solution by pipetting the appropriate amount of 1000 mg/L stock solution into a 100 mL volumetric flask and diluting to the mark with deionized water. Prepare the lower concentration working standards by diluting the highest concentration working standard with deionized water (Tables 2–4). Store standard solutions at 4 °C when not in use.

Table 2. Anion working standards.

	Concentration (mg/L)									
Acetate	0.02	0.04	0.1	0.2	0.4	1.0	2.0	5.0	12.5	25
Bromide	0.02	0.04	0.1	0.2	0.4	1.0	2.0	5.0	12.5	25
Chloride	0.4	0.8	1.6	4	8	16	40	80	200	400
Fluoride	0.02	0.04	0.1	0.2	0.4	1.0	2.0	5.0	12.5	25
Formate	0.02	0.04	0.1	0.2	0.4	1.0	2.0	5.0	12.5	25
Nitrate	0.02	0.04	0.1	0.2	0.4	1.0	2.0	5.0	12.5	25
Sulfate	0.02	0.04	0.1	0.2	0.4	1.0	2.0	5.0	12.5	25

Table 3. Working standards for the low concentration cations.

	Concentration (mg/L)						
Ammonium	0.05	0.1	0.25	0.5	1.0	2.5	5.0
Barium	0.25	0.5	1.0	2.5	5.0	10	25
Lithium	0.005	0.01	0.02	0.05	0.1	0.2	0.5
Potassium	0.1	0.2	0.5	1.0	2.0	5.0	10
Strontium	0.25	0.5	1.0	2.5	5.0	10	25

Table 4. Working standards for the high concentration cations.

	Concentration (mg/L)						
Calcium	2	4	10	25	50	100	200
Magnesium	0.5	1	2	5	12.5	25	50
Sodium	5	10	25	62.5	125	250	500

Sample Preparation

Samples were filtered with 0.2 µm polyethersulfone (PES) syringe filters (Thermo Scientific P/N 42213-PS). Samples were then diluted as indicated with 18 MΩ-cm resistivity degassed deionized water.

It is important to use 18 MΩ-cm resistivity deionized water for sample preparation as well as eluent and autosampler flush solutions to avoid system contamination, decreased sensitivity, and poor calibration. Degassing the deionized water by vacuum filtration prior to use is a good practice.

Results and Discussion

While a microbore IC system was used to determine anions and a capillary IC system was used for cation determinations, all of the data in this Application Note could have been collected using either system. Additionally, a dual pump Dionex ICS-5000⁺ IC system could have been used, but two separate systems were chosen to demonstrate the applicability of the methods described to both a high-end modular and a mid-level integrated system.

Anion Method

The method was optimized to obtain baseline separation (Rs USP >1.5) of the early eluting anions over the entire calibration range. Fifteen mM KOH was used for the first three min and a gradient of 15–29 mM KOH was used from three to four min. The eluent concentration was then held at 29 mM KOH for the remainder of the separation. Analyte peaks were well resolved (> Rs 1.5) with all anions eluting within 11 min (Figure 1).

Method Linear Calibration Ranges

To determine the linear calibration ranges, the peak responses to concentration were determined using triplicate injections of calibration standards (Table 2). While 10 calibration solutions were prepared for anion determinations, the two highest concentrations were only used for chloride, which was the most concentrated anion present in samples. Plotting peak area versus concentration demonstrated linearity for the concentration ranges used (Figure 2). Coefficient of determinations (r^2) ranged from 0.9990 to 0.9999 (Table 5).

Table 5. Anion calibration ranges and fitting results.

	Concentration (mg/L)	r^2
Fluoride	0.02–5.0	0.9995
Acetate	0.02–5.0	0.9996
Formate	0.02–5.0	0.9996
Chloride	0.4–400	0.9999
Bromide	0.02–5.0	0.9990
Sulfate	0.02–5.0	0.9990
Nitrate	0.02–2.0	0.9991

n = 3

Column:	Dionex IonPac AG18/AS18 columns, 2 mm i.d.
Eluent Source:	Dionex EGC KOH cartridge
Gradient:	15 mM KOH (0–3 min) 15–29 mM KOH (3–4 min) 29 mM KOH (4–12 min) 15 mM KOH (12–17 min)
Flow Rate:	0.25 mL/min
Inj. Volume:	2.5 µL
Column Temp.:	30 °C
Detection:	Suppressed conductivity, Dionex AERS 500 suppressor, recycle mode
Sample:	A: Water B: Standard

Peaks:	B	Resolution (R_S USP)
1. Fluoride	2.0 mg/L	1.7
2. Acetate	2.0	2.2
3. Formate	2.0	9.5
4. Chloride	80	14.7
5. Carbonate	—	
6. Bromide	2.0	3.2
7. Sulfate	2.0	2.3
8. Nitrate	2.0	

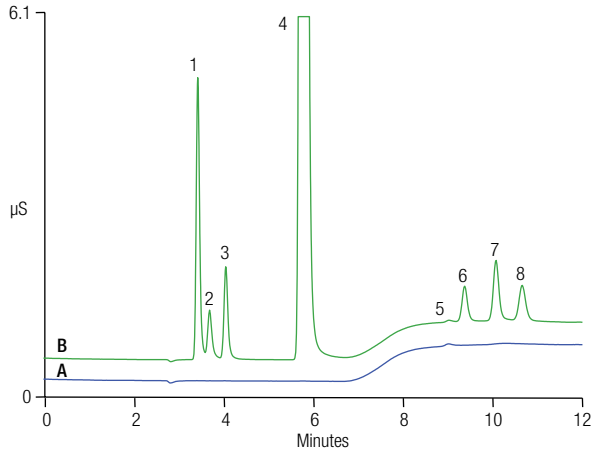


Figure 1. Separation of inorganic anions and organic acids using a KOH gradient.

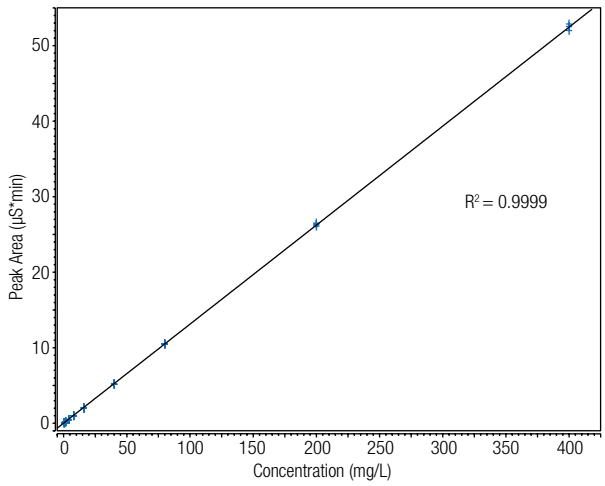


Figure 2. Chloride calibration curve.

Determination of Anion Concentrations in Produced Water

Produced water samples were diluted 50-, 200-, and 500-fold for the TX, CA, and ND samples, respectively, to be within the calibration range and to ensure that the column was not overloaded (Figure 3). The predominant anion present in produced water samples was chloride, followed by bromide at ~200-fold lower concentration, and then sulfate. Acetate and formate were present at less than 50 mg/L, while low, but detectable amounts of fluoride were only present in the TX sample (Table 6).

Table 6. Total anion concentrations in produced water.

	Concentration (mg/L)		
	Texas	California	North Dakota
Fluoride	3.9	—	—
Acetate	44	4.0	24
Formate	3.5	11	29
Chloride	8,000	52,000	190,000
Bromide	33	26	850
Sulfate	20	110	180

The anion concentrations in produced water varied considerably depending on its source with ND having the highest overall values, followed by CA, and then TX. Large differences are also evident when the produced water samples are compared to the ion concentrations that were obtained from Marcellus Shale flowback water (AN139).[5] For this comparison, fraction 10 was used because it had the highest ion concentrations for the majority of the anions quantified. Chloride was highest in ND, followed by the Marcellus Shale flowback sample, CA, and TX (Figure 4). It was anticipated that produced water samples would have considerably higher ion concentrations than flowback water because produced water contains a high proportion of formation brine (i.e. water naturally residing within the shale layer). In contrast, flowback can contain a significant amount of fracking fluid, which is typically low in salts. Consistent with this expectation, sulfate was significantly higher in produced water compared to flowback. While the chloride concentration from the ND sample was almost twice as high as for the Marcellus Shale flowback, the chloride concentrations of the other produced water samples were much lower. The reason for this discrepancy is likely due to comparing flowback and produced water from different fracking locations. It has been reported that total dissolved solids from different shale formations can vary by as much as an order of magnitude,[7] with considerable variation being found even within shale formations.[8]

Column:	Dionex IonPac AG18/AS18 columns, 2 mm i.d.	Detection:	Suppressed conductivity, Dionex AERS 500 suppressor, recycle mode
Eluent Source:	Dionex EGC KOH cartridge		
Gradient:	15 mM KOH (0–3 min) 15–29 mM KOH (3–4 min) 29 mM KOH (4–12 min) 15 mM KOH (12–17 min)	Sample:	A: Standard Produced water, filtered, 0.2 µm, diluted B: 50-fold dil. TX C: 200-fold dil. CA D: 500-fold dil. ND
Flow Rate:	0.25 mL/min		
Inj. Volume:	2.5 µL		
Column Temp.:	30 °C		

Peaks:	A	B	C	D
1. Fluoride	0.4 mg	0.078	—	—
2. Acetate	0.4	0.88	0.020	0.048
3. Formate	0.4	0.070	0.057	0.058
4. Chloride	16	160	260	380
5. Carbonate	—	—	—	—
6. Bromide	0.4	0.66	0.13	1.7
7. Sulfate	0.4	0.39	0.54	0.36
8. Nitrate	0.4	—	—	—

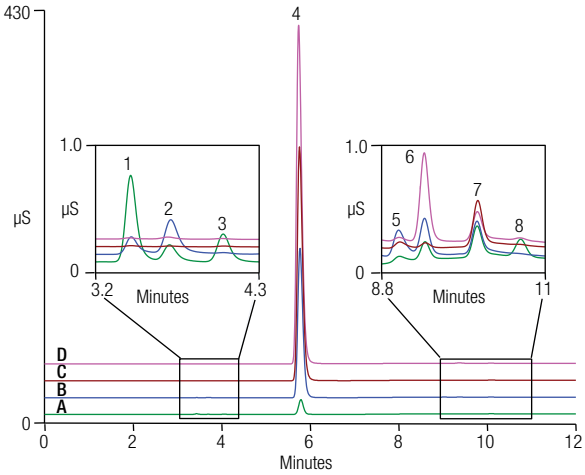


Figure 3. Determination of anions in produced water.

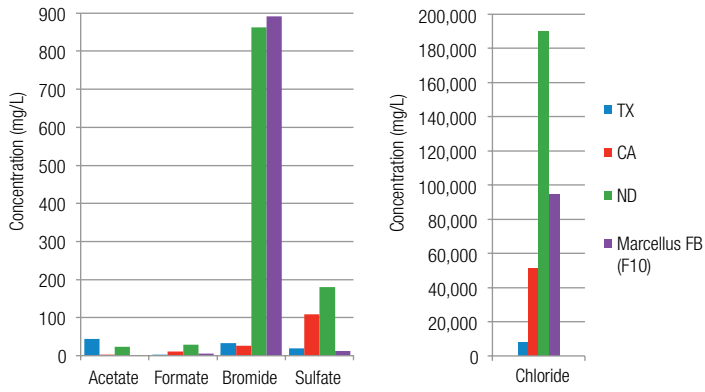


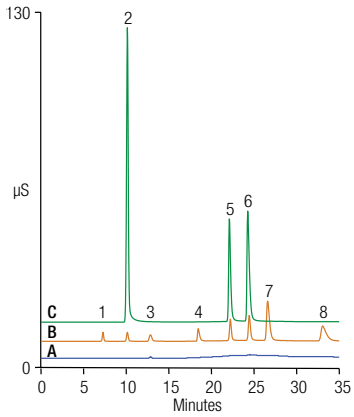
Figure 4. Comparison of anion concentrations in produced and flowback water.

Unfortunately, a direct comparison of flowback and produced water from the same site was not possible as part of this Application Note due to the lack of the appropriate samples. Another factor that can influence results is the initial quality of the water used for fracking. Treated wastewater is increasingly being used as a component of hydraulic fracturing fluid to reduce the impact on local water resources. As a consequence, the resultant flowback will have significantly higher salt concentrations compared to fresh water.

The high chloride concentration of the ND sample indicates that this water would need considerable treatment and/or dilution if it is to be used for additional fracking, whereas the TX water would require much less treatment. As with Marcellus shale flowback, the relatively high bromide concentration of the ND sample points to the need for monitoring of wastewater that is treated for surface water discharge, particularly if this water will be a source of drinking water production, due to the potential for bromate formation during ozonation or by surface water exposure to sunlight.

Cation Method

While the Dionex ICS-2100 RFIC system can be configured with a standard bore (4 mm i.d) or microbore (2 mm i.d.) Dionex IonPac CS16 column and equivalent results obtained,[6] for cation analysis, a capillary IC system was used to take advantage of the approximately 100-fold less water consumption (and waste generated) compared to a standard bore system. Using gradients of 20–30 mM MSA (0–10 min) and 30–55 (10–18 min) all analyte peaks were well resolved ((Rs USP) ≥ 3.5) and eluted within 35 min (Figure 5).



Column: Dionex IonPac CG16/CS16 columns, 0.5 mm i.d.
Eluent Source: Dionex EGC-MSA (capillary) cartridge
Gradient: 20–30 mM MSA (0–10 min)
30–55 mM MSA (10–18 min)
55 mM MSA (18–34 min)
20 mM MSA (34–40 min)
Flow Rate: 0.010 mL/min
Inj. Volume: 0.4 µL
Column Temp.: 40 °C
IC Cube Temp.: 15 °C
Detection: Suppressed conductivity, Dionex CCES 300 suppressor, recycle mode
Sample: A: Water
B: Low Conc. Stds
C: High Conc. Stds

Peaks:		B	C	Resolution (B) (R_s USP)
1.	Lithium	0.5 mg/L	—	8.8
2.	Sodium	2.0	25	6.2
3.	Ammonium	2.5	—	12.0
4.	Potassium	5.0	—	8.3
5.	Magnesium	2.5	5	4.5
6.	Calcium	5.0	10	3.5
7.	Strontium	25	—	6.7
8.	Barium	25	—	—

Figure 5. Separation of cations using an MSA gradient.

Method Linear Calibration Ranges

Plotting peak area versus concentration demonstrated linearity for the concentration ranges used (Figure 6). Coefficient of determinations (r^2) ranged from 0.9992 to 1.000 (Table 7). The exception was ammonium, which exhibited a quadratic fit relationship to concentration.

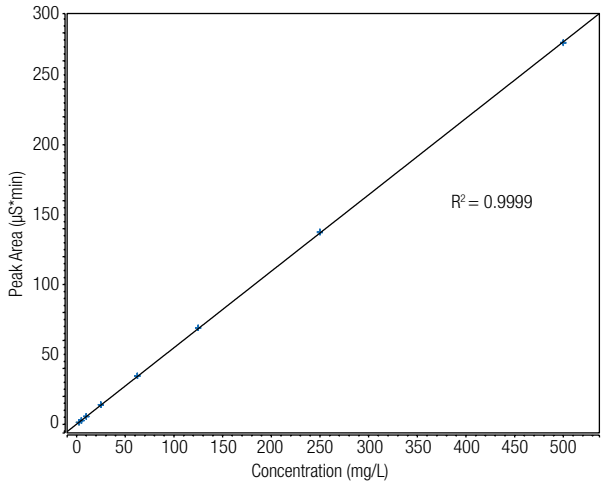


Figure 6. Sodium calibration curve.

Table 7. Cation calibration ranges and fitting results.

	Concentration (mg/L)	r^2
Lithium	0.005–0.5	0.9992
Sodium	5.0–500	0.9999
Ammonium*	0.05–5.0	0.9997
Potassium	0.1–10	0.9996
Magnesium	0.5–50	1.000
Calcium	2.0–200	1.000
Strontium	0.25–25	0.9995
Barium	0.25–25	0.9993

n = 3; * Quadratic curve fit

Determination of Cation Concentrations in Produced Water

As with anion analysis, the TX and CA samples were diluted 50- and 200-fold, respectively. The ND sample was diluted further from the 500-fold dilution to 1000-fold prior to analysis because the ammonium concentrations were higher than the linear range (Figure 7).

Consistent with the trend in anion concentrations, the concentration of cations was highest in the ND sample with sodium being the most abundant, followed by calcium, potassium, ammonium, and magnesium. Low concentrations of strontium and lithium were measured, while barium was not detected. A similar concentration trend was observed for the CA and TX samples, with sodium the highest, followed by calcium and magnesium. The cation concentrations present in the undiluted samples are shown in Table 8.

Table 8. Total cation concentrations in produced water.

	Concentration (mg/L)		
	Texas	California	North Dakota
Lithium	14.0	—	49
Sodium	4,300	28,000	92,000
Ammonium	50.0	18.0	3,700
Potassium	65.0	130	6,200
Magnesium	24.0	540	1,600
Calcium	500	2,200	21,000
Strontium	13.0	—	530

There was more than twice as much sodium (the most abundant cation) in the produced water from North Dakota than in the other samples, including the flowback (Figure 8). Marcellus flowback water had considerably higher concentrations of strontium and barium, comparable levels of lithium, magnesium, and calcium, and much lower amounts of ammonium and potassium when compared to the ND produced water sample. While strontium and barium were low or absent in produced water, the relatively high calcium concentrations in the ND sample make recycling more challenging due to its propensity to form scale. As noted for anions, the differences in cation concentrations were likely due to the differing geological compositions of the shale layers in which the hydraulic fracturing occurred.

Column:	Dionex IonPac CG16/CS16 columns, 0.5 mm i.d.	Detection:	Suppressed conductivity, Dionex CCES 300 suppressor, recycle mode
Eluent Source:	Dionex EGC-MSA (capillary) cartridge	Sample:	Produced Water, filtered, 0.2 µm, diluted
Gradient:	20–30 mM MSA (0–10 min) 30–55 mM MSA (10–18 min) 55 mM MSA (18–34 min) 20 mM MSA (34–40 min)		A: 50-fold dil. TX B: 200-fold dil. CA C: 1000-fold dil. ND
Flow Rate:	0.010 mL/min		
Inj. Volume:	0.4 µL		
Col. Temp.:	40 °C		
IC Cube Temp.:	15 °C		

Peaks:	A	B	C
1. Lithium	0.28 mg/L	—	0.049
2. Sodium	86	140	92.0
3. Ammonium	1.0	0.088	3.7
4. Potassium	1.3	0.64	6.2
5. Magnesium	0.48	2.7	1.6
6. Calcium	10.0	11.0	21.0
7. Strontium	0.25	—	0.53

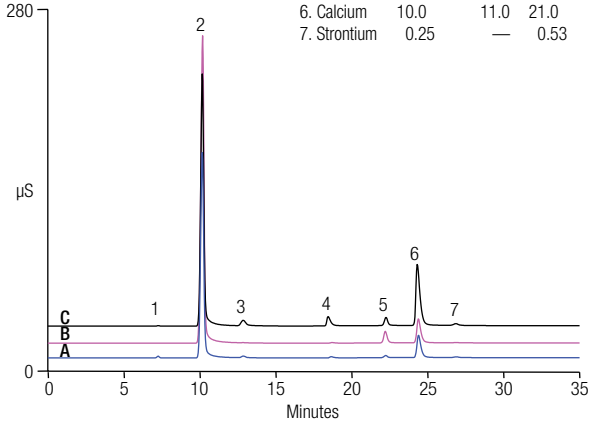


Figure 7. Determination of cations in produced water.

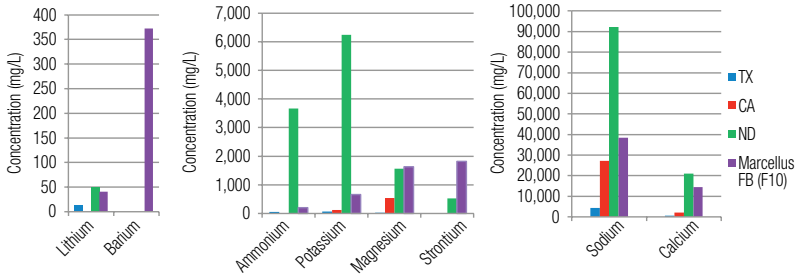


Figure 8. Comparison of cation concentrations in produced and flowback water.

Conclusion

This Application Note demonstrates that the concentration of both high and low abundance ions in hydraulic fracturing produced water can be accurately determined using the Dionex ICS-2100 IC system with a microbore Dionex IonPac AS18 column and a Dionex ICS-5000$^+$ HPIC system with a capillary Dionex IonPac CS16 column. The chromatographic conditions were optimized so that all of the analytes eluted within 35 min. The most abundant anions were chloride, sulfate, and bromide, while sodium, calcium, magnesium, potassium, and ammonium were the most prevalent cations. The ion concentrations of produced and flowback wastewater varied significantly. As with flowback water, planning wastewater treatment and reuse strategies of produced water should consider the high concentration of the scale-forming calcium in produced water and the potential for downstream conversion of bromide to bromate.

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